by rats, primarily in the urine and, to a lesser extent, through the feces. Preliminary data indicate phthalamic acid or a closely related water-soluble derivative to be the major metabolite of excretion, thus suggesting that in the rat, as in the cotton plant (15) and the steer (10), Imidan detoxication involves hydrolysis to water-soluble metabolites. In this respect, it follows a pattern similar to that established for other phosphate esters (1, 14).

Radioactivity remaining in tissues accounted for only 2.6% of the administered dose and no tissue selectively stored radioactive residues. The low fat residues is in contrast to findings with chlorinated hydrocarbon insecticides which accumulated in fat and is in accord with metabolic studies with several organophosphate insecticides (6, 7). Since there was a general reduction in radioactive residues in all tissues between the 72- and 120-hour intervals, it was not possible to assess fully whether the radioactivity found in the various tissues represented C^{14} metabolites which were incorporated into normal body constituents or whether the radioactive residues were only transiently stored and weakly bound to tissues.

Essentially no cleavage of the carbonyl carbon in the phthalimide moiety occurred in vivo, since no more than a trace of $C^{14}O_2$ was liberated. Faigle and coworkers (12) also found essentially

no $C^{14}O_2$ in the exhaled air from rats and dogs which were orally fed with Thalidomide-C^{14} (α-phthalimidoglutarimide) uniformly labeled in the carbonyl groups. Although the trace amount of $C^{14}O_2$ found in this study could possibly have been liberated from an impurity, it is suspected that it may represent bacterial disruption of the phthaloyl moiety either in the gastrointestinal tract or in the feces. Dagley and coworkers (11) reported that many bacteria were capable of cleaving benzoates and polycyclic aromatic compounds to yield $C^{14}O_2$.

Acknowledgment

The authors are grateful to J. B. McBain for determining solvent partition characteristics of urinary and fecal radioactivity, to J. Leach for technical assistance, both of this laboratory, and to G. LaRoche, Donner Laboratory, University of California, Berkeley, Calif., for loan of the digestion apparatus used in this study to combust the hide samples to CO_2.

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HERBICIDE ADSORPTION STUDIES

Adsorption and Leaching of Herbicides in Hawaiian Sugarcane Soils

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With 16 increments of water, each equivalent to 1 acre-inch, leaching was negligible below a depth of 2 inches in a sugarcane topsoil for diuron applied at experimental rates up to 20 pounds per acre. Monuron at rates up to 10 pounds per acre did not leach appreciably below 4 inches. The soil had a saturation adsorption limit for diuron of about 47 pounds per acre-inch when the diuron was applied as a nearly saturated aqueous solution. Leaching of these herbicides at usual field rates in normal sugarcane topsoils is a minor consideration; it is a major contributory factor to sugarcane injury in exposed subsoils. The equilibrium availability of the herbicide in a given soil solution depends on adsorption, herbicide concentration, and the soil-water ratio.

MOST Hawaiian sugarcane topsoils show an unusually high degree of physical adsorption of herbicides from aqueous solutions compared to most U. S. mainland soils (5, 11). Subsoils do not exhibit the same degree of adsorption. Two major factors are believed to

account for the ability of the topsoils to fix most of the applied herbicide: a relatively high "organic matter" content and a considerable quantity of carbon from the combustion of dry leaf trash and cane tops prior to crop harvest. The "organic matter" represents accum-

ulated organic residues and the decomposition products of 12 to 15 tons of sugarcane roots and variable quantities of leaf trash incorporated in the soil after each two-year crop cycle. In dry, irrigated areas (about 50% of the total acreage in sugarcane) analysis of "or-

ganic matter" by the usual wet oxidative methods will average 2.5 to 5%, increasing to 15 to 20% or more in the cultivated areas of high rainfall (7). Such oxidative analyses presumably do not account for the carbon (leaf charcoal) content. It thus would be difficult to assess the relative importance of the two adsorptive forces, except that the adsorptive capacity of leaf carbon is far in excess, about a factor of 10^3 to 10^4 on a weight basis, of the soil itself.

Our earlier experiments measured adsorption as an equilibrium between a quantity of soil and an excess of aqueous herbicide solution; the soil and solution were shaken together, and enough solution was removed for analysis of residual herbicide. For present purposes, this adsorptive measurement process is referred to as a static equilibrium because the values are fixed for a given soil-solution ratio and a given concentration of herbicide in solution.

The present paper is concerned with the measurement of adsorption in soil columns under leaching conditions. Most of the work reported concerns the adsorption of diuron, 3-(3,4-dichlorophenyl)-1,1-dimethylurea, on one soil (designated Kunia B, a red low humic Latosol, Molokai family) with a static adsorption capacity of 88% for the herbicide. For this soil the K value for diuron, measured at unit concentration from the Freundlich isotherm plot, was about 13. This soil, taken from a dry, surface-irrigated field, represents the largest single soil series under sugarcane cultivation in Hawaii. At 15 atm. this soil contains 21.3% water (approximate wilting point); at 4 atm., 23.4%; and at 0.33 atm., 29.5% (approximate field capacity).

Experimental Procedure

Leaching and Profile Distribution of Diuron Solution. Diuron was added as a water solution of 17.28-p.p.m. concentration, equivalent to 5.7 pounds per acre at 1.5 acre-inches of water, to the top of air-dry soil columns 1, 2, 4,

Table I. Initial Percolation of 17.28-P.P.M. Diuron Solution in Dry Soil Columns

| | | Acre-Inches | | |
Soil Depth, Inches	Diuron solution added	Water added	Total percolate obtained	Diuron in Percolate, P.P.M.
1	1.5	0	0.75	1.6
2	1.5	0	0.22	0.0
3	1.5	1.25	0.22	0.0
4	1.5	2.25	0.35	0.0

and 6 inches in depth. To give an initial percolate from all tubes, the 4- and 6-inch columns received an additional 1.25 and 2.25 acre-inches of water, respectively. Analysis of the initial percolate is shown in Table I. After the initial wetting and drainage, the columns received 10 increments of 0.75 acre-inch each of water with no lapse of time between increments; each increment was collected separately and analyzed for diuron.

The 4-inch column was divided into 1-inch segments; these portions and the soil from the 1-inch soil column were analyzed for diuron (Table II). Soil segments were analyzed by the standard colorimetric procedure. Blanks for soil analysis without diuron were used as corrections where necessary.

Soil Saturation with Diuron Solution. Successive 2.5-acre-inch increments of an aqueous solution containing 11.52 p.p.m. of diuron were added to a 1.2-inch soil column until the percolate concentration of the herbicide was equal to that in the input solution. The input solution was then changed to an aqueous solution containing 23.04 p.p.m. of diuron; this resulted in further adsorption until a new saturation was reached. The input solution was again changed to one containing 34.56 p.p.m. of diuron, and the incremental addition was continued until saturation was reached. Since the last concentration was near the practical saturation limit of diuron in water, the soil contained the maximum amount of chemical possible under the conditions used. The 1.2 acre-inches of soil adsorbed 47 pounds of

Table II. Distribution of Adsorbed Diuron in Soil Columns after 10 Increments of 0.75 Acre-Inch Each of Water

| Soil Depth, Inches | Adsorbed Diuron | | | |
| | 1-Inch Column | | 4-Inch Column | |
	%	Lb.	%	Lb.
1	67	3.8	69	4.0
2	...	...	31	1.7
3	...	...	0	0
4	...	...	0	0
Total percolates	33	1.9	0	0
Recovery	100	5.7	100	5.7

Table III. Saturation Limit of a Soil for Diuron as Determined by Percolation

| Diuron Input Solution | | Saturation Limit, Lb./1.2 Acre-Inch Soil |
Concn., p.p.m.	No. of 2.5-inch increments to saturate	
11.52	15	28
23.04	7	40
34.56	3	47

diuron from the near-saturated solution. Table III shows the data for each concentration.

Desorption of Diuron from a Saturated Soil. The soil column, saturated with 47 pounds of diuron per 1.2 acre-inch, was successively leached with increments of 2.5 acre-inches of water; each increment required about 15 minutes to drain through the soil layer. Analysis of the incremental leachates was performed using untreated soil leachates as reference.

Both the adsorption (preceding section) and desorption phases of the experiment are shown in Figure 1. Desorption removed a total of 78% of the applied herbicide in 17 increments; the first nine increments removed 70% of the saturated quantity, and the next eight increments removed a further 8%. The recovery data are shown in Figure 2.
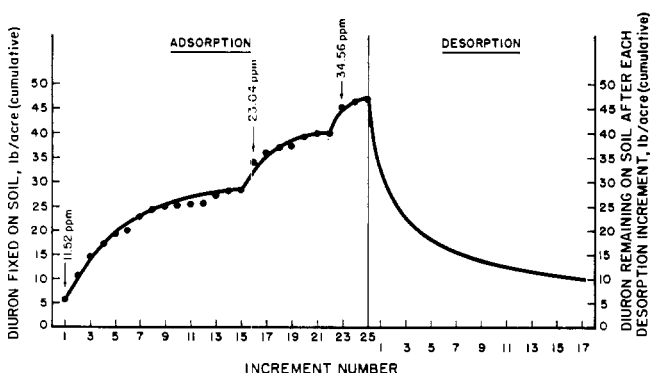

Figure 1. Saturation of a soil with aqueous diuron solutions and desorption of diuron with water
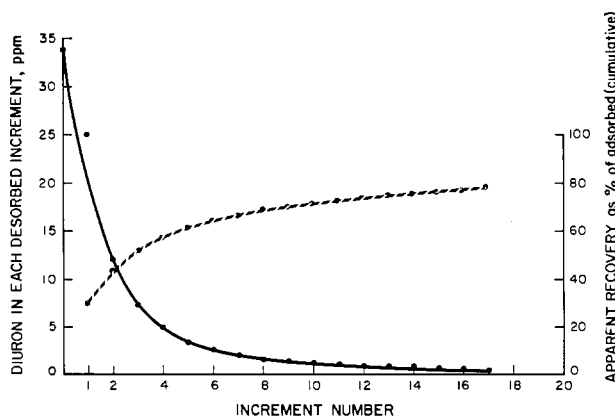

Figure 2. Recovery of diuron from a saturated soil

— Concentration in each 1-inch increment of water
——Cumulative percentage of adsorbed herbicide

Leaching of Diuron from Applications of Powered Herbicide. Diuron powder, equivalent to 5, 10, 20, and 40 pounds per acre, was placed on the surface of 4-inch soil columns, which were then just wetted with an initial 3 acre-inches of water. Sixteen increments of 1 acre-inch each of water were added, the percolates from each increment were collected separately and were analyzed for diuron. Finally the columns were divided into 1-inch segments, and each segment was analyzed for residual diuron (Table IV).

Only the rate of 40 pounds per acre resulted in the leaching of diuron below 4-inch levels, at the following concentrations:

Increment No.	Diuron, P.P.M.
1–6	0
7	0.25
8	0.30
9	0.36
10	0.52
11	0.60
12	0.72
13	0.98
14	0.98
15	1.10
16	1.22

Dissolution of Monuron by Percolating Water. Powdered purified crystals of monuron at 9.0 pounds per acre were applied to the surface of a layer of pure silica sand in a filter tube. Five increments of 2 acre-inches each of water were allowed to percolate through the tubes. Analysis of the increments showed:

Increment	Amount Dissolved, %	Monuron Concn., P.P.M.
1	47	8.9
2	21	4.0
3	8	1.6
4	4	0.8
5	3	0.5
Total	83	

Leaching of Monuron on Soil Columns. Monuron solution at 12.50 p.p.m. was added at an equivalent of 11.70 pounds per acre to two soil columns, one 2 inches and the other 5.5 inches in depth. A single increment of 4 acre-inches of water was applied to each column; the columns were sectioned, and each segment and the leachate was analyzed (Table V).

In another experiment, a monuron solution equivalent to 2.75 pounds in 2 acre-inches of water was shaken with an equivalent of 1-acre-inch of air-dry soil until the solution had equilibrated. Then the slurry was poured into a filter tube, and the liquid was allowed to drain. A total of 78% of the monuron was adsorbed by the soil (2.15 pounds), 8.2% remained in the adhering soil solution (0.75 acre-inch at 1.37 p.p.m., 0.22 pound), and 13.8% drained (1.25 acre-inches at 1.37 p.p.m., 0.38 pound).

Five increments, 1 acre-inch each of water, were added successively to the column, and the percolates were analyzed as 1.23 p.p.m. (0.27 pound per acre), 1.04 (0.23), 0.92 (0.20), 0.69 (0.15), and 0.59 (0.13). The remaining soil column

Table IV. Distribution of Diuron in 4-Inch Soil Columns (from Dry Powder and Solution Applications) after 16 Increments of 1 Acre-Inch of Water

		Diuron Recovered, Lb./Acre				
Soil Depth, Inches	Solution, 5 lb./acre		Dry Powder			
		5 lb./acre	10 lb./acre	20 lb./acre	40 lb./acre	
1	2.8 ⎫94%	4.0 ⎫100%	7.2 ⎫93%	11.5 ⎫90%	15.0 ⎫73%	
2	1.9 ⎭	1.0 ⎭	2.1 ⎭	6.5 ⎭	14.2 ⎭	
3	0.2	0	0.5	1.8	6.7	
4	0.04	0	0.2	0.2	2.5	
Total percolates	0	0	0	0	1.6	

Table V. Monuron Leaching on Soil Columns

	Monuron Recovery			
	2-Inch Column		5.5-Inch Column	
Column Segment	%	Lb.	%	Lb.
1st inch			68	7.8
Top 0.5 inch	43	5.4		
Lower 0.5 inch	27	3.2		
2nd inch			23	2.7
Top 0.5 inch	11	1.3		
Lower 0.5 inch	8	0.9		
3rd inch			4	0.5
4th inch			1	0.1
5th inch			2	0.2
Last 0.5 inch			2	0.2
Leachate total	11	1.3	0	0

was sectioned into two 0.5-inch segments and analyzed:

	Amount Retained	
	%	Lb./acre
Top 0.5 inch	10.5	0.20
Bottom 0.5 inch	40.0	1.10
All leachates	49.5	1.36

Static Desorption of Monuron and Diuron by Dilution. This procedure (11) is based on a serial dilution with water after adsorption had been established on several soils. Dilution was made in 100-ml. increments from 100 to 1000 ml. based on 50 grams of air-dry soil and 100 ml. of herbicide solution used to establish the initial adsorption equilibrium. The data for 10-fold dilution for four topsoils and one subsoil are shown in Table VI. The dilution necessary to effect substantially total recovery of diuron was estimated by extrapolation of the lines obtained by plotting log dilution against desorption, assuming that desorption remains linear (Figure 3).

Results and Discussion

Optimum sugarcane growth requires considerable water. Ideally, in most Hawaiian soils, 2.5 acre-inches of water at intervals of about 14 days, or about 6 inches per month, approximately matches the evapotranspiration losses averaged over the year, totaling at least 65 inches per year or 130 per 2-year crop cycle. In practice, furrow-irrigated areas receive nearly 300 acre-inches of water per crop to ensure adequate field coverage. Unirrigated areas receive a minimum of 70 inches of rainfall per year, reasonably uniformly spaced, and
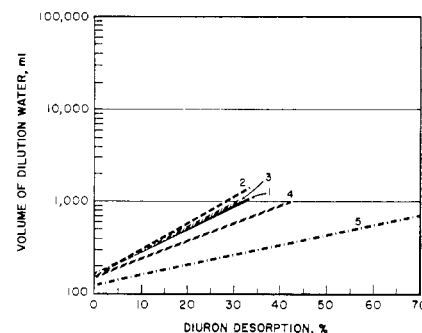

Figure 3. Desorption of diuron from five soils by serial dilution

Refer to Table VI for soil properties

may receive up to a maximum average of about 180 inches per year or 360 inches per crop. Water in excess of evapotranspiration is essentially lost by surface and internal drainage.

The diverse condtitons make it difficult to predict a pattern of soil-applied herbicide performance or to define "typical" areas. The more biologically active and relatively nonselective herbicides such as monuron, diuron, simazine, atrazine, and ametryne (2-ethylamino-4-isopropyl-amino-6-methylmercapto-s-triazine) perform reasonably well in most areas at 3 to 6 pounds per acre without crop injury, although the normal 30-to-60-day control of annual weeds to be expected at these rates is short compared to most mainland U. S. conditions. Crop injury from herbicides applied to soil has usually been associated with exposed subsoils or areas of thin eroded soil. The normal 18-inch plowing depth produces reasonable uniformity of structure and composition, with rather abrupt transitions to the substructure.

Dissolution of the finely divided wettable-powder herbicides in water has been found to be rapid and would be expected to be complete in the first few inches of applied water. This is in agreement with the view of Hartley (3). Assuming that water was present in excess of saturation, dissolution rate should be a function of the surface area of the undissolved particles. Monuron concentration in successive 2-inch increments of water leached through a sand column was found to decrease at approximately a linear rate when the concentration was plotted as a semilog function against the amount of water; the first 2-inch increment dis-

Table VI. Desorption of Monuron and Diuron from Five Soils by Serial Dilution

Soil	Soil Family	Initial Adsorption, % Monuron	Initial Adsorption, % Diuron	Recovery at 10-fold Dilution, % Monuron	Recovery at 10-fold Dilution, % Diuron	Extrapolated Total Recovery (Dilution Factor), Diuron
1. OSRS topsoil	Mixed	75	90	46	34	500
2. Kah-5 topsoil	Gray hydromorphic	77	92	49	30	1200
3. Hilo-540 topsoil	Hydrol humic latosol	74	88	41	31	600
4. Hilo-560 topsoil	Hydrol humic latosol	68	83	57	42	125
5. Hilo-560 subsoil	Hydrol humic latosol	20	36	60	89	13

solved about one-half the total herbicide as the following data indicate. About 4.5 pounds of diuron should dissolve in 0.5 acre-inch of water to form a saturated solution at 40 p.p.m. An experiment showed that about one fourth of this amount dissolved during filtration of 0.5 acre-inch of water through the powdered herbicide.

Although contact time between water and powdered herbicide is important and depends on the amount of water applied, infiltration rate, field capacity, and evaporation-rate loss from the soil surface, the amounts of water used on sugarcane are sufficient to dissolve all of the commonly used herbicides.

Published adsorption and leaching studies of monuron and diuron emphasize the wide diversity of experimental conditions associated with a variety of soils, crops, water, and assay of residual herbicide. There is general agreement that, with a given amount of herbicide and water, adsorption and leaching are most closely correlated with organic matter (4, 6-11). Other properties with varying degrees of correlation measured by either adsorption or growth measurements are cation exchange capacity (8), clay content (8), clay type (2), pH (2, 8, 11), phosphate fixation (11), or other factors (8). Diuron apparently leaches less readily than monuron, although no direct comparisons were found in the literature. Disappearance under field conditions appears to take place in situ rather than as the result of leaching out of the profile. Evaporation had no special effect on leaching of monuron, but more frequent water applications caused greater leaching than less frequent applications (9). Sherburne, Freed, and Fang (7) concluded that the depth of monuron leaching was inversely related to soil moisture prior to start of leaching and that leaching increased with increasing water percolation.

It seems unfortunate that two methods have been used in the literature to measure "residual" herbicide, a situation which leads to some confusion. Some authors (6, 8, 10), using bioassay of a sensitive test plant, measured available herbicide rather than total residual. Ogle and Warren (6) found greater residual toxicity in a sandy soil than in a muck soil; yet they concluded correctly that the sandy soil was most highly leached. The retained herbicide con-

tributed greater toxicity in the sandier soil because of its lower equilibrium adsorption. Other authors (4, 5, 7, 9, 11) have used chemical assay which gave total chemical residue, with perhaps some hydrolytic metabolites and natural soil constituents; yet they established little about the residual phytotoxicity of the soil layers.

In the present work, the initial percolation of a diuron solution at 17.28 p.p.m. through a soil column over 1 inch in depth resulted in the complete removal of the diuron from the soil. Subsequent addition of 10 increments of 0.75 acre-inch of water did not remove any detectable herbicide from the soil columns over 2 inches in depth; the retained herbicide remained in the upper 2 inches, with two thirds of the total in the upper inch. Reversible adsorption theory assumes that 10 increments of extracting liquid will remove 40% of the retained herbicide which has a 95% equilibrium retention and will leave 60% on the soil (0.95^{10}) (3, page 130). Initial percolation in the soil left 92.8% on the 1-inch column; the 10 water increments (assuming 95% adsorption) should have removed 40% of that or 37.1%, leaving about 55.7%. The actual figure of 67% retained (25.8% removed) indicates that the soil adsorption under the particular set of conditions, was higher than 95%— nearly 97%. The difference between this figure and the static adsorption value of 88% can best be accounted for by the change in soil-water ratio from 1 to 2 for the static conditions to nearly 1.0 to 0.75 in the columns.

The "saturation" of this soil with diuron solutions of several concentrations revealed that, based on a 1.2-inch column, 28 pounds per acre could be adsorbed from an 11.52-p.p.m. solution, 40 pounds from a 23.04-p.p.m. solution, and 47 pounds from a nearly saturated 34.56-p.p.m. solution. These saturation figures are probably characteristic of a particular soil and represent the maximum retention from a constant source of supply.

The leaching or desorption of diuron from this saturated soil was of interest because, again after any given number of increments of leaching water, the retained herbicide should be characteristic of the soil. This soil retained 13.5 pounds after 10 increments of water;
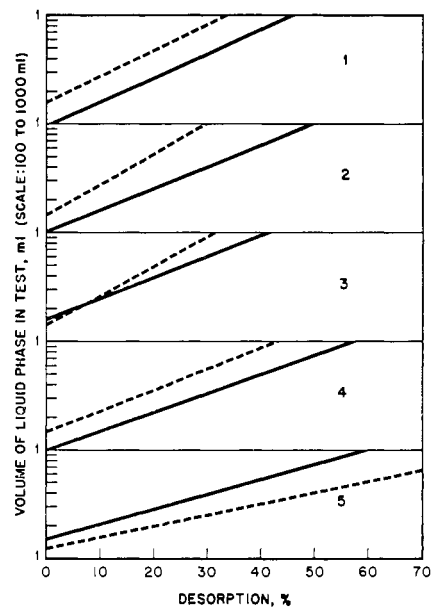

Figure 4. Desorption of monuron and diuron from five soils by serial dilution

Refer to Table VI for soil properties
— — — Diuron
——— Monuron

at 97% retention, another 10 increments would be expected to remove 26.5% $[100 - (0.97^{10}) 100]$, or 3.5 pounds more, to a lower layer. A total of 90 increments should remove 90%, or all but 1 pound. An extrapolation of the curve in Figure 1 indicates nearly complete removal at 75 increments.

These figures represent one set of experimental conditions and do not take into account any effects of evaporation, time between water increments, and chemical and biological decomposition of the herbicide. It seems obvious, however, that at field rates of less than 10 pounds per acre, no appreciable leaching can be expected in this soil and similar ones below the 2-inch level before the chemical disappears from other causes. Using the approximate figure of 97% adsorption for the soil at a given moisture level, after each irrigation or rainfall which brings the soil to that moisture level, the retained herbicide will re-equilibrate with the soil solution to provide 3% in solution, which is presumably available to the root system of seedling plants. When the total residue in the top few inches of soil decreases sufficiently by microbial or chemical action, or by

plant uptake so that the 3% equilibrium concentration does not result in a phytotoxic soil solution for one or more species, control of seed weed species will presumably be lost. The remaining herbicide will no longer be effective, especially under the semitropical conditions of continuous seed germination. The soil-water ratio is very important. In fact "adsorption" is not a static property characteristic of a given soil, but is a condition of equilibrium which exists among soil, water, and herbicide and depends on the relative ratios of each component. The "adsorptive capacity" of a soil for a herbicide may vary from essentially 100% to essentially zero, depending on the amount of water present and the herbicide concentration in the water.

Leaching of monuron was not studied in detail. A single increment of 4 inches of water through a soil column, after monuron had been applied at 11.7 pounds per acre as a solution, resulted in about 90% retention in the top 2 inches of the soil column; about 70% was retained in the top inch and 20% in the second inch. In a different experiment with monuron, about 50% was retained in the top inch after five increments of 1 inch each of water had been added.

Further evidence of the difference between monuron and diuron desorption in several soils was obtained by a static serial-dilution technique. Recovery of the two herbicides is shown in Figure 4 as a semilog function of the soil-solution ratio from 1:4 to 1:20 in dilution steps of 1:8, 1:12, 1:17, and 1:20. We do not know the reason for the reversal of desorption in the one subsoil in which diuron was desorbed more easily than monuron.

The effect of application rate and use of powdered herbicide rather than solutions was studied with diuron at 5, 10, 20, and 40 pounds per acre. The effect of dissolution from the powder was particularly noticeable. After 16 increments of 1 acre-inch each of water, 80.4% of the 5-pound rate was retained in the top inch; 55% was retained in a comparison column using diuron solution at an equivalent 5 pounds per acre. As Table IV indicates, there was no diuron in the percolates below 4 inches except at the 40-pound rate. The ratio of diuron retained in the top inch to that in the second inch decreased from 4 to 1 at 5 pounds per acre to 1.06 to 1 at 40 pounds per acre. The percentage of diuron that appeared below the first inch increased from 20% at 5 pounds per acre to 62.6% at 40 pounds per acre. The percentage increase was roughly linear with the amount of herbicide. The percentage that appeared below the second inch was zero at 5 pounds, 0.7 at 10 pounds, 2.0 at 20 pounds, and 10.8 at 40 pounds.

The percolate analyses indicated tha the 40-pound rate was still showin leaching below 4 inches after 16 inche of percolating water.

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HERBICIDE RESIDUES

Determination of 3-Amino-1,2,4-triazole Residues in Sugarcane

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3-Amino-1,2,4-triazole applied as an interline herbicide spray solution on young sugarcane at rates up to 40 pounds per acre leaves residues of less than 0.002 p.p.m. by the time of harvesting at the end of 2 years. Detailed improvements in the analytical method, especially in instrumentation, permit measurement of less than 0.1 µg. of amitrole in sugarcane or in concentrated samples of sugarcane juice.

AMITROLE (3-amino-1,2,4-triazole) has been found useful for the control of certain perennial grass and broadleaf weeds common in sugarcane fields and on irrigation ditch banks. The control of certain strains of Bermuda grass (*Cynodon dactylon*), wing-leaved passion vine (*Passiflora pulchella*), and *Panicum* and *Digitaria* spp. was of special interest.

Application of directed sprays in sugarcane produced a moderate amount of chlorosis in the leaves, which persisted for 3 to 4 weeks without appearing to affect yields or subsequent growth. A study was designed to determine the residual amitrole in sugarcane after several applications at excessive rates over the period of the crop cycle (2 years) or until disappearance of the amitrole could be assured. One application of 5 pounds (active) per acre with one possible repeat treatment at the same rate constituted the optimum practical and economic use; the study was based on 5, 10, and 20 pounds per acre with one repeat treatment at the same rate after 8 weeks, making total treatments of 10, 20, and 40 pounds per acre.

Two tests were conducted, one at the Kilauea Sugar Co. on the island of Kauai under surface, furrow-irrigated, in an area of 50 inches of rainfall per year, and one at the Hilo Sugar Co. on the island of Hawaii under non-irrigated conditions, with rainfall of about 150 inches per year.

Experimental

The two tests were begun in December 1961 in sugarcane $2^{1}/_{2}$ months old on four replicate, $^{1}/_{100}$-acre plots, with additional 20 × 40 foot replicate plots